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Zrak na delovnem mestu - Določanje celotnih organskih izocianatnih skupin v zraku z uporabo 1-(2-metoksifenil)piperazina in tekočinske kromatografije

Workplace air - Determination of total organic isocyanate groups in air using 1-(2-methoxyphenyl)piperazine and liquid chromatography

Air des lieux de travail - Dosage des groupements isocyanates organiques totaux dans l'air par dérivatisation avec la 1-(2-méthoxyphényl)pipérazine et par chromatographie en phase liquide

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Workplace air — Determination of total organic isocyanate groups in air using 1-(2-methoxyphenyl) piperazine and liquid chromatography

Air des lieux de travail — Dosage des groupements isocyanates organiques totaux dans l'air par dérivatisation avec la 1-(2-méthoxyphényl)pipérazine et par chromatographie en phase liquide

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

This third edition cancels and replaces the second edition (ISO 16702:2007), which has been technically revised.

The main changes are as follows:

— the references have been reviewed and updated, and obsolete references have been removed or replaced where appropriate;

— the Introduction and the Scope have been revised to conform with current ISO editorial requirements and to improve clarity on the applicability of the method;

— minor technical changes have been made, including the harmonization of common names of chemical compounds;

— additional information has been added on the use of impingers for sampling isocyanates (Clause 4);

— a note on the requirement to record the final volume of toluene has been introduced (8.8.2);

— examples of suitable internal standards have been added (9.9).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Isocyanates are organic compounds containing the isocyanate functional group (-NCO) and are widely used in industrial applications such as the manufacture of polyurethanes, paints, coatings, foams, plastics and adhesives. They are highly reactive substances and are well-established respiratory sensitisers, representing a major cause of chemically induced occupational asthma. Occupational exposure can occur primarily by inhalation and, potentially, by skin contact.

Occupational exposure limits for isocyanates have been established in several countries and are generally expressed in terms of total isocyanate, defined as the sum of monomeric and polymeric isocyanates, including oligomeric and prepolymeric forms. These limits are specified as long-term (8 h time-weighted average) and short-term (15 min) exposure limits and require analytical methods capable of determining total isocyanate groups in workplace air.

The sampling and analysis of airborne isocyanates present particular challenges due to the diversity of chemical structures, molecular masses and physical forms encountered in workplace atmospheres. Isocyanates can be present as vapours, aerosols or liquids and often occur as mixtures of monomers, oligomers and polymers. Other airborne substances, such as water vapour, dust, amines and alcohols, can also be present and can interfere with analytical determination. In addition, reference standards for many polymeric isocyanates are currently not available, despite their contribution to total isocyanate exposure.

Because of the high reactivity of the isocyanate functional group, workplace air monitoring is commonly based on derivatisation techniques that convert isocyanates into more stable compounds suitable for analysis. The method described in this document is based on derivatisation with 1-(2-methoxyphenyl) piperazine (MP), forming stable urea derivatives that are analysed by liquid chromatography with electrochemical and ultraviolet and visible detection. The analytical approach was established[1] using widely used industrial diisocyanates, such as methylene diphenyl diisocyanate (MDI), hexamethylene diisocyanate (HDI), and toluene diisocyanate (TDI), together with their associated oligomeric and polymeric forms. It has subsequently been applied to a broader range of organic isocyanates containing free isocyanate groups e.g. phenylisocyanate (PI), isophoronediisocyante (IPDI), naphthyldiisocyante (NDI), hydrogenated methylene diphenyl diisocyanate (H12MDI) and butylisocyanate.

If both isocyanates and amines are present and need to be determined, a standard capable of simultaneous determination of both classes of compounds can be more appropriate (see ISO 17734-2[2]). This method has also been modified for the determination of monoisocyanates produced during thermal degradation,[3] for the use of mass spectrometric detection,[4] and for alternative sampling equipment, such as 37 mm filters and other filter holders; however, these modifications are not covered by this document. When using a modified version of this method, it is the responsibility of the user to demonstrate the validity of the modifications.

Sampling approaches depend on the physical form of the isocyanates present. Filters are suitable for the collection of vapour-phase isocyanates, while combined impinger and filter arrangements are used for aerosol sampling. The method has been applied to commonly occurring mono- and diisocyanates and to polymeric isocyanates derived from these monomers.

Workplace air — Determination of total organic isocyanate groups in air using 1-(2-methoxyphenyl)piperazine and liquid chromatography

1 Scope

This document specifies a test method for the sampling and analysis of airborne organic isocyanate (NCO) compounds in workplace air. The method covers organic compounds containing free isocyanate functional groups, including monomeric, oligomeric, prepolymeric and polymeric isocyanates, and addresses the measurement of total isocyanate groups in air samples collected for the assessment of occupational exposure.

The method is suitable for personal air sampling in the breathing zone for the determination of time-weighted average concentrations over sampling periods ranging from approximately 10 min to 8 h, although it can be applied to shorter sampling periods with high isocyanate air levels. It can also be used for background or fixed-location air sampling; however, due to aerodynamic effects, samplers designed for personal sampling do not necessarily exhibit the same collection characteristics when used for other purposes. It covers the measurement of airborne organic isocyanates over a concentration range of approximately 0,1 $\mu g/m^3$ to 140 $\mu g/m^3$ for a nominal air sample volume of 15 l; under the conditions specified in this document, typical qualitative and quantitative detection limits correspond to approximately 0,07 $\mu g/m^3$ and 0,3 $\mu g/m^3$, respectively, for a 15 l air sample.

This document does not apply to the simultaneous determination of isocyanates and amines, nor to modified methods employing alternative sampling devices or detection techniques not described in this document.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 13137, *Workplace atmospheres — Pumps for personal sampling of chemical and biological agents — Requirements and test methods*

ISO 18158, *Workplace air — Terminology*

EN 1540, *Workplace exposure – Terminology*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18158, EN 1540 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
bulk isocyanate material
isocyanate (3.3) based product supplied in bulk form and used in a manufacturing or processing operation

3.2
diisocyanate
chemical compound with two *isocyanate* (3.3) functional groups

3.3
isocyanate
chemical compound with one or more isocyanate (nitrogen carbon oxygen) functional groups

3.4
monomer
chemical compound that joins with other identical compounds to form dimers, trimers, *oligomers* (3.5) or polymers

Note 1 to entry: Classes of isocyanate monomers include: monoisocyanates, containing one isocyanate functional group, e.g. methyl isocyanate; *diisocyanates* (3.2), e.g. di(4-isocyanatophenyl)methane (MDI); and triisocyanates, e.g. tri(4-isocyanatophenyl)methane.

3.5
oligomer
compound of low relative molecular mass with multiple *isocyanate* (3.3) functional groups, formed by the combination of isocyanate *monomers* (3.4)

3.6
prepolymer
isocyanato-terminated reaction product of a *diisocyanate* (3.2) or polyisocyanate with a stochiometric deficiency of a hydroxyl-terminated polyol

Note 1 to entry: When used in the workplace, these prepolymers then react further to form polyurethanes or similar compounds

3.7
uncertainty
parameter, associated with the result of a measurement, that characterises the dispersion of the values that can reasonably be attributed to the measurand

Note 1 to entry: The parameter can be, for example, a standard deviation (or a given multiple of it), or the width of a confidence interval.

Note 2 to entry: Uncertainty of measurement comprises, in general, many components. Some of these components can be evaluated from the statistical distribution of the results of series of measurements and can be characterised by standard deviations. The other components, which can also be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information. This is often referred to as type A and type B evaluations of uncertainty, respectively.

4 Principle

The selection of the sampling arrangement in this method is determined by the physical state of the isocyanate being monitored. When sampling isocyanate aerosols, air is drawn through a glass impinger containing a solution of 1-(2-methoxyphenyl)piperazine (MP), followed by an MP-impregnated backup filter. In contrast, for isocyanates present in the vapour phase, sampling may be carried out using an MP-impregnated filter alone.

Impingers provide more efficient collection than filters when isocyanates are present in the aerosol phase. Nevertheless, very fine particles, particularly those smaller than 1 µm, might not be fully retained by an impinger,[5,6] and a so a treated downstream filter is required.[7] Sampling methods based solely on filters can lead to underestimation of exposure where larger particles (greater than 10 µm) are present or under

conditions of high aerosol loading, as the capacity of the derivatising reagent can be exceeded.[8] To address this limitation, filters are extracted on site using a solution of the derivatising reagent.

A known volume of air is passed either through the MP solution in the glass impinger with an MP-treated filter downstream (for aerosols) or directly through an MP-impregnated filter (for vapours). Any organic isocyanates collected react with the MP reagent to form stable, non-volatile urea derivatives. The resulting sample extract is then concentrated and analysed using high-performance liquid chromatography (HPLC) with ultraviolet and visible (UV) and electrochemical (EC) detection. Peaks attributable to isocyanate derivatives are identified based on their combined UV and EC responses, supported by diode array detection (DAD) spectral library matching, mass spectrometric confirmation where available, and comparison with derivatised bulk isocyanate material,[9] see 9.8.1.

Where reference standards of MP-derivatised isocyanates are available (e.g. HDI, MDI, and TDI isomers), quantification is performed using UV detection. In cases where appropriate standards are not available, such as for isocyanate oligomers, prepolymers or polymers, EC detection is used for quantification, with calibration based on the corresponding monomer standard. The overall airborne isocyanate concentration is obtained by summing all peaks attributed to isocyanate.

5 Reagents

5.1 MP reagent [1-(2-methoxyphenyl)piperazine]. This reagent is commercially available with a purity of >98 % by mass.

5.2 Toluene, of a purity suitable for HPLC.

NOTE Toluene (5.2) is commonly used as the reagent solvent, it must be free from compounds co-eluting with the substance(s) of interest. Before use for the preparation of MP-impregnated filter (8.3.2) or for preparation of monomer standards (7.2.1), it is advisable to dry the solvent with anhydrous calcium chloride or magnesium sulfate.

5.3 Acetonitrile, of a purity suitable for HPLC.

5.4 Methanol, of a purity suitable for HPLC.

5.5 Hexane, of a purity suitable for HPLC.

5.6 Dichloromethane, of a purity suitable for HPLC.

5.7 Water, of a purity suitable for HPLC, conforming with ISO 3696 Grade 1.

5.8 Anhydrous sodium acetate, of recognized analytical grade.

5.9 Acetic anhydride, of recognized analytical grade.

5.10 Glacial acetic acid, of recognized analytical grade.

6 Apparatus

Before sampling and analysis, clean (9.2) all glassware, including impingers (6.4). Usual laboratory apparatus, and in particular the following, shall be used.

6.1 Sampler. The choice of sampler used depends on the form in which the isocyanate is present. For vapour phase isocyanates, sampling can be carried out using an MP-impregnated filter (8.3.2) only. For mixtures of airborne particles and vapour, the use of an impinger (6.4) backed by an MP-impregnated filter is recommended.

6.2 Filter. Filters of diameter 25 mm are suitable for use in the selected sampler. The chosen filter type should be suitable for collection of stable samples of isocyanate and have a capture efficiency of not less than 0,95, see 9.10 and Annex A. A glass fibre filter impregnated with 1,2-MP (5.1) reagent (8.3.2) is suitable, e.g. Whatman GF/A[1)] or equivalent.

6.3 Filter holder. Details of suitable sampling heads are given in MDHS14/4.[16] A 25 mm Institute of Occupational Medicine (IOM) head[1)] fitted with a stainless-steel cassette is recommended for filter samples. For aerosol sampling using the impinger (6.4) and filter (6.2) combination, it has been found to be more convenient to use the 25 mm Swinnex[1)] filter holder.

6.4 Midget impinger. Several designs of bubblers and impingers are available.[10,11] A midget impinger consists of a graduated receiver and a tapered inlet tube.

NOTE "Non-spill" impingers are commercially available.

6.5 Sampling pump, which shall fulfil the requirements of ISO 13137 or equivalent. Specific safety regulations can apply.

6.6 Tubing, plastic, rubber or other suitable material, approximately 900 mm long, of appropriate diameter to ensure a leak-proof fit to both pump (6.5) and sample tube or tube holder, if used. Fluoroelastomer or similar tubing has been found to have fewer problems due to extraction of contaminants associated with it. It is not recommended to use any tubing upstream of the first collection element [filter (6.2) or impinger (6.4)] as sample losses can occur.

6.7 Flowmeter, portable, capable of measuring the appropriate flow rate to within ±1 %, and calibrated against a primary standard.[12] Flowmeters incorporated in sampling pumps (6.5) are not suitable for accurate measurement of the flow rate. However, they can be useful for monitoring the performance of samplers, provided they have adequate sensitivity.

6.8 Filtration equipment. A solvent resistant filter unit of < 0,5 µm pore size for filtration of LC solvents. Syringeless filters or < 0,5 µm syringe filters for filtration of the desorbed samples prior to LC analysis.

6.9 Ancillary equipment.

6.9.1 Belts or harnesses, to which the sampling pump (6.5) can be conveniently fixed, unless the pump is sufficiently small to fit into a worker's pocket.

6.9.2 Flat-tipped tweezers for handling the filters.

6.9.3 Protective holder for impinger (6.4).

6.9.4 Charcoal trap to protect the sampling pump (6.5) from toluene vapour (if plastic pumps are being used).

6.10 Liquid chromatograph. An HPLC (6.10) linked to UV and EC detectors is required. The EC detector should be used in the oxidation mode. A DAD is also advisable for confirmation of identification. Temperature fluctuations must be avoided to obtain the sensitivity required in this method. This can be achieved by fitting the HPLC column and EC detector with a thermostat. EC performance can be improved by recirculating the mobile phase in a closed loop and by use of a guard cell (set to approximately 50 mV above analytical cell potential) before the injector. A pulse dampener will also decrease the LC system noise (pulse ripple) and so increase signal to noise ratio.

1) GF/A, IOM and Swinnex are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.